

# Auger depth profiling of heat-treated Au/Ni thin films

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Gold/nickel thin films heat treated in the temperature range 200–500 °C, have been analysed for composition–depth profiles using Auger electron spectroscopy in combination with simultaneous Ar<sup>+</sup> ion sputtering. The present investigation indicates that pronounced nickel diffusion through the gold layer takes place due to annealing at 500 °C for 5 min, whereas a noticeable nickel diffusion through the gold layer needs 3 h of annealing at 200 °C. The measured concentration profiles suggest diffusion mechanisms involving infinitely fast grain boundary diffusion coupled with defect-enhanced diffusion into the grains. Copyright © 2002 John Wiley & Sons, Ltd.

**KEYWORDS:** gold; nickel; depth profiling; diffusion

## INTRODUCTION

Auger electron spectroscopy (AES) is one of the most powerful materials analysis techniques available today, and many reviews of this technique have been written.<sup>1–4</sup> In AES, the atomic core levels are ionized by an incident electron beam at kiloelectron-volt energies and the subsequent radiationless Auger transition and escape of the Auger electron provide elemental identification through energy analysis of the emitted electron. This technique is surface sensitive because of the limited mean free path of the Auger electrons, typically in the range of 4–20 Å.<sup>5</sup> This makes AES ideally suited for examining thin surface layers. Depth profiles of the chemical composition can be obtained by simultaneously ion milling the surface and examining it with AES.

For reasons of conductivity, adherence and reliability, conductor systems used in thin-film circuits normally consist of multilayer structures of more than one metal.<sup>6</sup> Among these, gold/nickel (Au/Ni) thin films are most frequently used in the microelectronics industry. Gold is known to have low resistivity, good corrosion resistance and good bondability. However, the adhesion of Au to most substrate materials is poor. Therefore, an intermediate metal such as Ni is deposited first to improve the adhesion between the substrate and the Au.<sup>7</sup> To manufacture circuits and to allow for reworking of the product, a variety of sophisticated interconnection schemes are utilized, such as soldering, wire bonding and brazing. However, all these operations normally are performed at elevated temperature (200–500 °C),<sup>8</sup> where diffusion may occur and degrade the performance of the circuit. In the present work, Au/Ni thin films annealed at the fabrication temperature range were analysed using AES in combination with Ar<sup>+</sup> ion sputtering to investigate the effect of heat treatment on the element distribution

with depth in this bilayer system. It is intended that these data can be employed to define appropriate processing temperatures.

## EXPERIMENTAL

Silicon (111) wafers were highly cleaned<sup>9</sup> and used as substrates. Pure Ni (99.99%) and pure Au (99.99%) were evaporated sequentially in a vacuum (single pump-down) to a thickness of 200 nm for each layer as determined by a quartz crystal gauge. The substrate temperature was 100 °C. The ultimate pressure in the thermal evaporation system was  $2 \times 10^{-6}$  mbar with a pressure of  $1 \times 10^{-5}$  mbar being typical during deposition. Deposited samples were heated in a vacuum furnace of  $4 \times 10^{-6}$  mbar pressure to various temperatures between 200 and 500 °C for times between 5 and 180 min.

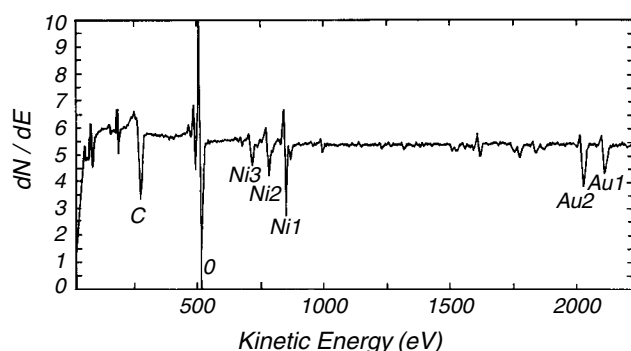
Composition–depth profiles for annealed samples were obtained using AES in combination with *in situ* Ar<sup>+</sup> ion sputtering. The Auger system used was a SAM 660 scanning Auger microprobe manufactured by Perkin-Elmer and operated at the following specifications and conditions: for the primary electron beam the current was 500–600 nA, the incident energy was 5 keV and the beam width was 1 µm rastered across an area of  $\sim 100 \times 100$  µm<sup>2</sup>; for the Ar<sup>+</sup> ion beam the voltage was 3 kV, the current was 0.8 µA, the spot size was 800 µm, scanned during sputtering in an area of  $3 \times 3$  mm<sup>2</sup>, the basic vacuum in the analysis chamber was  $3 \times 10^{-10}$  mbar and during sputtering it was  $2 \times 10^{-8}$  mbar. The mean grain size of the investigated films was determined from x-ray diffraction analysis<sup>10</sup> using a Philips PW 1710 diffractometer. The x-ray tube was operated at 40 kV voltage and 30 mA current, using monochromated Cu K $\alpha$  radiation of 0.15406 nm in wavelength. The surface morphology and microstructure of some samples were examined using a JEOL scanning electron microscope, model JSM-6400.

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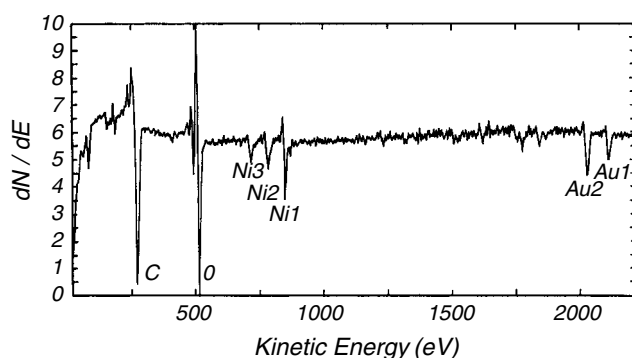
## RESULTS AND DISCUSSION

Auger survey spectra were recorded in differential mode at the surface of each heat-treated specimen. A typical spectrum is shown in Fig. 1 for a specimen annealed at 500 °C for 5 min. The presence of carbon and oxygen peaks can be attributed easily to exposure of the specimens to air before Auger analysis. Nickel peaks (at 848, 783 and 716 eV) are seen in the surface survey spectrum, in spite of Ni being the inner layer. This provides evidence that heat treatment at 500 °C for a few minutes causes a significant diffusion of Ni atoms through the Au layer to the surface. The transfer of Ni from beneath the Au layer to its outer surface also was seen for the specimens annealed at 400, 300 and 200 °C, as Fig. 2 demonstrates for a specimen annealed at 200 °C for 180 min. By comparing Fig. 1 and Fig. 2 it can be concluded that the magnitude of diffusion due to annealing at 500 °C for 5 min is greater than that caused by annealing at 200 °C for 180 min, as can be inferred from the peak-to-peak heights of Ni in these two figures.

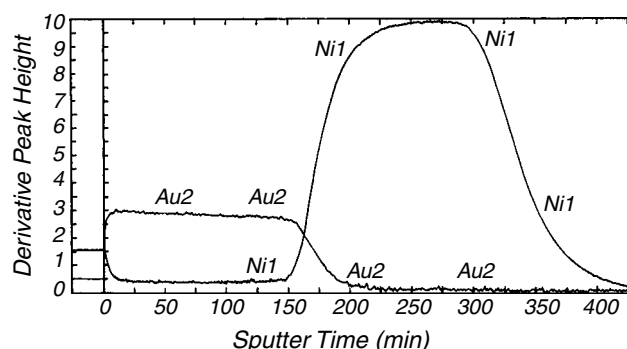
By simultaneous Ar<sup>+</sup> sputtering and AES analysis, the elemental signal intensity (peak-to-peak height) as a function of the sputtering time was obtained, as shown typically in Fig. 3 for a specimen annealed at 500 °C for 5 min. The 848 eV Ni peak (Ni1) and the 2024 eV Au peak (Au2) were adopted in our analysis because they are the highest peaks, as Figs 1 and 2 indicate, and this improves the precision of the calculations. Converting the measured Auger peak heights to concentrations,<sup>2,11,12</sup> the atomic percentage concentrations of Ni and Au as a function of sputtering time and consequently



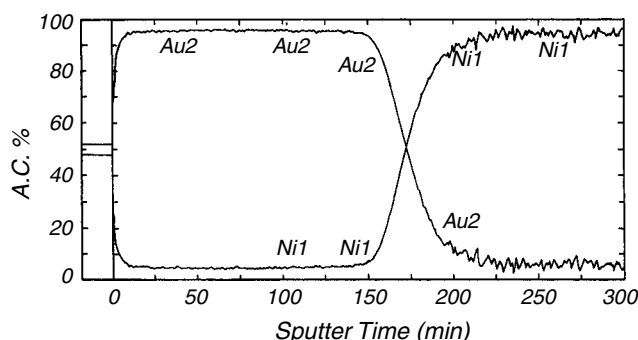
**Figure 1.** The AES surface survey spectrum of Au/Ni thin films annealed at 500 °C for 5 min.



**Figure 2.** The AES surface survey spectrum of Au/Ni thin films annealed at 200 °C for 180 min.



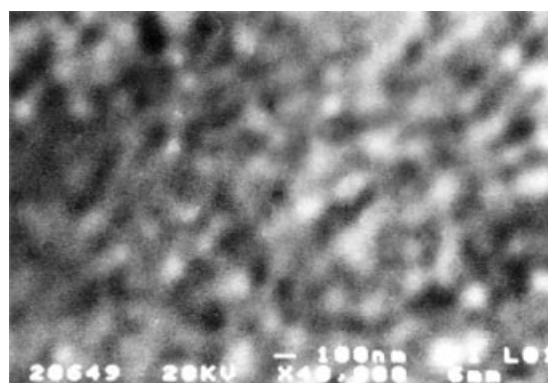
**Figure 3.** Derivative Auger peak heights of Au and Ni versus sputter time for a specimen annealed at 500 °C for 5 min.



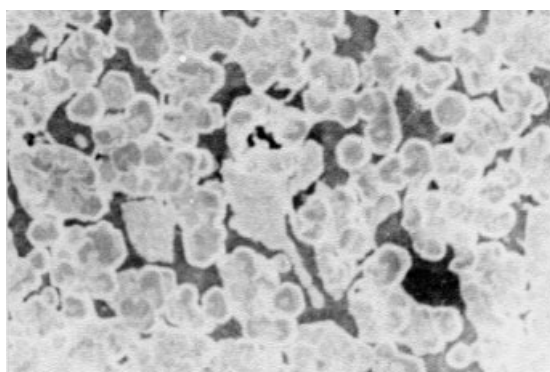
**Figure 4.** Atomic percentage concentrations of Au and Ni versus sputter time for a specimen annealed at 500 °C for 5 min.

as a function of depth were obtained as shown in Fig. 4 for a specimen annealed at 500 °C for 5 min. As is obvious from this figure, Ni has diffused through the Au layer and its atomic concentration reaches a level of almost 6 at.%. The Ni diffusion was observed also in the specimens annealed at 400, 300 and 200 °C but at lower levels of atomic concentration in spite of increasing the annealing time, e.g. annealing at 300 °C for 80 min causes Ni diffusion through Au at a level of 1.5 at.%. Therefore, during the manufacturing processes of Au/Ni thin-film devices, which are normally performed in the temperature range 200–500 °C, it is advisable to avoid the highest temperatures in this range as far as possible in order to minimize the amount of Ni diffusion to the Au surface, which may degrade the performance of the device.<sup>13</sup> On the other hand, Fig. 4 shows that Au atoms also have diffused through the Ni layer to the substrate, causing loss of adhesion, as was observed experimentally for the specimen annealed at 500 °C for 5 min. The roughness of the curves in Fig. 4 after a long sputtering time can be attributed to the distortion effects<sup>11,12,14</sup> caused by energetic Ar<sup>+</sup> ion (3 keV) sputtering.

One obvious feature of the diffusion profile in Fig. 4 is the presence of essentially a zero concentration gradient of the Ni in the Au layer. This leads to the plausible assumption that grain boundary diffusion is extremely fast compared with the lattice diffusion. Rapid grain boundary diffusion means that the boundaries are constantly full of the diffusant (Ni) and the slower lattice diffusion causes a concentration gradient just near the interface. Geometrically, grain boundaries represent



(a)



(b)

**Figure 5.** Scanning electron micrographs of Au/Ni thin films: (a) as deposited; (b) annealed at 500 °C.

a small fraction of the total volume, given roughly by  $\delta/r$ , where  $\delta$  denotes the effective boundary width ( $\sim 0.5$  nm) and  $r$  denotes the mean grain radius.<sup>15</sup> In the Au film,  $r$  was determined from x-ray diffraction analysis to be  $\sim 57$  nm and the grain boundaries constitute only 0.9% of the total volume. Even if it is assumed that a maximum of 50 at.% solute can be accommodated within the boundaries, the grain boundary diffusion can account only for  $\sim 0.5$  at.%, which is a small portion of the 6 at.% Ni measured in the Au layer after annealing at 500 °C for 5 min. The remainder must, therefore, result from additional diffusion into grain interiors. The lattice diffusion coefficient is estimated to be  $7.5 \times 10^{-14} \text{ cm}^2 \text{ s}^{-1}$  at 500 °C from extrapolation of bulk data at higher temperatures.<sup>16</sup> The correspondingly small diffusion distance of  $2(Dt)^{1/2} \sim 95$  nm clearly indicates that

this mechanism is not sufficient to account for the measured Ni concentration. An intragranular mechanism involving enhanced diffusion via high defect density, such as the pin holes displayed in Fig. 5, must therefore make a substantial contribution. Hence, the measured concentration profiles represent the composite result of defect-assisted lattice diffusion fostered by accelerated grain boundary diffusion.

## CONCLUSION

From the data of the present work it can be concluded that the interdiffusion mechanisms needed to interpret the measured concentration profiles involve rapid grain boundary diffusion coupled with defect-enhanced diffusion into the grains. It is noted that a pronounced Ni diffusion through the Au layer takes place due to annealing at 500 °C for 5 min, whereas a noticeable Ni diffusion through the Au layer needs 3 h of annealing at 200 °C.

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